XAS study of thermally induced nano-structuring in metal-doped carbon films

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Introduction

Carbon is a widely used material for protection against mechanical, thermal and particle loads. Doping often ameliorate the properties of carbon layers, e.g. for wear protection coatings or carbon-based facing elements in fusion devices. The reactivity of carbon against hydrogen species is the main drawback for the fusion application due to lifetime shortening of the components (chemical erosion) and formation of hydrocarbon layers, which is of safety concern if radioactive tritium is used as a fuel for fusion. Doping with carbideforming transition metals reduces chemical erosion considerably. In order to elucidate the underlying mechanism, properties of doped carbon films have to be characterized down to atomic scale.

Methods and Materials

We present systematic investigation of amorphous carbon films doped with different metals (V, Ti, Zr, W) at concentrations up to 20 at% produced by dual-source magnetron sputter deposition. Their composition, homogeneity and phase were characterized for as-deposited and annealed films using X-ray diffraction, electron microscopy and Rutherford backscattering [1]. Local atomic environment of the metal species and their evolution upon concentration and annealing has been studied using X-ray Absorption Spectroscopy at the V,Ti K-edges and W L_{2.3}-edges at the A1 and E4 beamlines of HASYLAB/DESY.

Results and Discussion

Position and shape of the absorption edges indicate that all the metals are in non-metallic state already after deposition. As an example we present the results of $C_{0.87}V_{0.13}$ layer study, Fig.1. The V K-edge of 'as deposited' layer is shifted towards higher energy from that of V metal. Further chemical shift and evolution of the spectral shape with increasing annealing temperature indicates evolution of the local structure around vanadium to that of vanadium carbide (VC). Similarly, EXAFS modulations evolve with temperature to those measured in carbide. Fourier transformed EXAFS spectrum in the 'as deposited' layer reveal first peak at the distance characteristic for carbide, whereas it amplitude and ratio to the second neighbor peak reveals amorphous surrounding of the deposited vanadium. After annealing the two separated peaks, originating from the carbon and vanadium neighbors, are pronounced. Their shape and amplitude evolve to the that of vanadium carbide already after annealing to 1100 K. This result is fully consistent with XRD measurements, which revealed carbide nano-crystals of about 5 nm diameter [1]. Although a evolution of the local structure around metal species to that of carbidelike is observed in all the systems studied, the characteristic

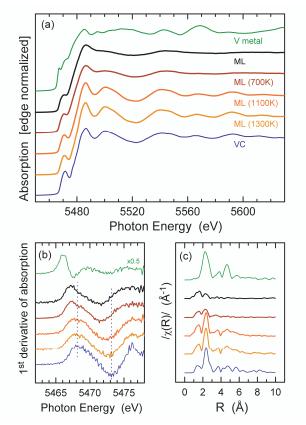


Fig. 1. (a) Vanadium K-edge XAS spectra of the 'as deposited' $C(220nm)/C_{0.87}V_{0.13}(280nm)/C(320nm)$ multilayer (ML) as well as annealed in 700K, 1100K and 1300K. Reference spectra of V metal and VC are shown for comparison. (b) First derivative of the corresponding XANES spectra. (c) Fourier transform of the corresponding EXAFS spectra.

temperatures and final carbide structures are different, which helps in better understanding of their erosion properties.

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[1] M. Balden, B. Cieciwa, I. Quintana, E. de Juan Pardo, F. Koch, M. Sikora, B. Dubiel, Surf. Coat. Technol. **200**, 413-417, (2005).